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(54) Concentration apparatus

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## PATENT SPECIFICATION

### 1. TITLE OF THE INVENTION

Concentration apparatus

### 2. CLAIM

- (1) Concentration apparatus wherein a pressurizing and heating section (A) for concentration liquid, and a concentration chamber (B) which is equipped with a spray section for heated and pressurized concentrating-solution, pressure control unit for the concentration chamber, are connected with a pipeline.

- (2) Concentration apparatus which is described in Claim 1 and characterized that the pressurizing and heating section (A) is made to have a structure that is able to convert the evaporation latent heat of the liquid in the concentrating-liquid into sensible heat of the solution of the concentrating-liquid.
- (3) Concentration apparatus which is; a concentration apparatus wherein a pressurizing and heating section (A) for concentration liquid, and a concentration chamber (B) which is equipped with a spray section for heated and pressurized concentrating-solution, pressure control unit for the concentration chamber, are connected with a pipeline; and is characterized that it is integrated with an analyzing unit for the concentrated-liquid and integrated with a control system that controls pressure and temperature of the pressurizing and heating section (A) and pressure of the concentration chamber using the results of analysis.
- (4) Concentration apparatus which is described in Claim 3 and characterized that the composition analyzing unit for the concentration liquid is a line viscosity meter.
- (5) Concentration apparatus which is described in Claim 3 and characterized that the pressurizing and heating section (A) is made to have a structure that converts the evaporation latent heat of the liquid in the concentrating-liquid into sensible heat of the solution of the concentrating-liquid.

### 3. DETAILED DESCRIPTION OF THE INVENTION

This invention concerns an apparatus that concentrates low concentration solution into high concentration solution, and especially it concerns an concentration apparatus that is effective for concentrating medium viscosity solution and which is multi-component mixture solution.

There have been various types of apparatuses developed as concentration apparatuses of liquid and their characteristic is that structure of apparatuses are different depending on the viscosity of liquid to be concentrated, and single axis evaporation unit, dual axes evaporation unit, and thin film cascade type evaporation unit, etc. have been known as for the apparatus that concentrate high viscosity concentrating-liquid, and evaporation kettle, multi-efficiency kettle, etc. have been known as for the apparatus that concentrate low viscosity concentrating-liquid. However, for the ones with medium viscosity around 1 to 1,000 poise of viscosity of concentrating-liquid, there are many ones having their own behavior being very complicated and hard to handle, and therefore, efficient and versatile concentration apparatus has not been developed, and current situation is that there is a need to measure such as property, flow behavior, etc. of individual concentrating-liquid and individually develop apparatus that meets each concentrating-solution.

Therefore, the inventors have discovered that concentrated-liquid is relatively easily obtained by using a method to spray concentrating-liquid, wherein evaporation latent heat of components to evaporate in the concentrating-liquid is converted into solution sensible heat of the concentrating-liquid, into a concentration chamber that is kept at low temperature and low pressure, in an investigation targeting a development of a concentration apparatus which is able to efficiently concentrate concentrating-liquid of medium viscosity and has versatility, and discovered that the apparatus that can be effectively used for this method becomes

the most effective apparatus by making it in a structure that maintains heating and pressurizing section and the concentration chamber for the concentrating-liquid in separate condition, and completed this invention.

The summary of this invention is that it is a concentration apparatus which is equipped with a concentration apparatus that connects a pressurizing and heating section (A) for concentrating-liquid and a concentration chamber (B) which is equipped with a spraying section of concentrating-liquid, a pressure control unit for the concentration chamber, a temperature control unit for the concentration chamber, a volatile component outlet and a concentrated-liquid outlet, with a pipeline, and a composition analyzing device for the concentrated-liquid, and this analyzing unit is connected to the pressurizing and heating section (A) and/or the pressure control device of the concentration chamber with an relay to control temperature and pressure of concentrating-liquid and pressure of the concentration chamber.

Figure 1 is a schematic drawing of typical concentration apparatus of this invention. In Figure 1, 1 is concentrating-liquid supply line, 2 is concentrating-liquid pressure feed pump, and using this pump, the concentrating-liquid is kept under pressurized. 3 is concentrating-liquid pressurizing and heating section, which is an apparatus to put evaporation latent heat for volatile components that are to be removed from the concentrating-liquid in the concentrating-liquid as sensible heat. As the heating device of this heating unit, use of device that is able to quickly heat such as slit heater, onlater, etc., for example, is desired. 4 is a spray section that expands the diffusion area of the concentrating-liquid that has been integrated with the evaporation latent heat of the volatile components and to have the volatile components that self boils leave without resistance, and it is desired to be constituted with such as nozzles, slits, etc., in order to make the concentrating-liquid into mist. 5 is concentration chamber, 6 is concentration chamber temperature control unit, 7 is volatile component outlet, and 8 is volatile component condensation section. 9 is pressure control valve that controls vacuum level of the condensation chamber and 10 connects to its vacuum source. 11 is concentrated solution outlet, 12 is a takeout pump for the concentrated solution, and 13 is a component analyzing device for the concentrated-liquid. 14 is a control system for controlling the composition of the concentrated solution by controlling the vacuum level of the concentration chamber with the analysis result of the composition of the concentrated solution. 15 is a control system for controlling the composition of concentrated solution by controlling heating temperature of the concentrating section\* with the analysis result of concentrated solution. The concentration chamber 5 is possible to be attached with a mixing blade or a scraping blade, however, it is not especially necessary because the heating section and the evaporation section are separated in this apparatus and there is hardly any deposition on the wall of the concentration tank.

*\* Translator's note: This "section" shall be a mistake of "liquid" or "solution" both of which are mixed used for meaning the same thing in this Japanese document.*

As the concentrated solution analyzer used in this invention, it is able to use various types of such as physical and chemical analyzers, however, the most convenient one is an infrared analyzer which is convenient for analyzing volatile components, and as a physical means it is a viscosity meter.

Attention should be paid when concentrating the concentrating-liquid using the apparatus of this invention that the substance contained in the concentration solution would not cause density pattern such as scorching or gelation at the heating surface when heating and pressurizing the concentrating-liquid, and that is to convert the evaporation latent heat of the volatile component of the concentrating-liquid into the sensible heat of the concentrating-liquid at the heating and pressurizing section and to have it evaporate when the concentrating-liquid is sprayed in the concentration chamber.

Because it is difficult to apply such operations with previously developed concentration apparatuses, the undesirable concentration distribution occurs during its concentration process, however, in case of using the apparatus of this invention above described problem would not occur because the heating and pressurizing section (A) and the concentration chamber for the concentrating-liquid are separated, and further it is able to significantly shorten its concentration time compared to using previous apparatuses.

As the materials that can be most effectively concentrated with the concentration apparatus of this invention, it is able to be used for cases such as; concentration by removal of ethylene glycol at the initial polymerization of polyester, concentration by removal of water from rubber compound containing water, concentration by removal of water from water containing polymer that is obtained by water solution polymerization, and concentration when making spinning liquid from waste fiber in wet spinning process; and especially for production of solvent solution of polymer that is concentrated by evaporating a part of water and solvent for water containing polymer after adding solvent.

In this case, it is necessary to select the pressure and temperature of the concentration chamber (B) so that when the heated and pressurized concentrating material is sprayed, its volatile component evaporates in boiling condition, for example.

The characteristics of the functionality of this concentration apparatus are such as;

- (1) it is able to integrate necessary amount of heat for evaporating the volatile component in the concentrating-solution ahead of time as the sensible heat of the solution,
- (2) it uses the heat capacity that has been integrated as the sensible heat of concentrating-solution for evaporation latent heat of the volatile component, and it is equipped with a small-hole spraying section such as nozzle or slit so that sufficient diffusion surface is obtained for effective diffusion and removal of the volatile component when the concentration liquid is sprayed under low temperature and low pressure and the volatile component self boils,
- (3) because the heating function and the evaporation function are completely separated and the heating section and the evaporation section are separately constituted, there is no generation of foreign substance in the concentrating material,
- (4) because the evaporation time is minimum, it is able to do accurate control in short time,
- (5) because it is able to provide grate evaporation area, it is able to control evaporation amount only with heating temperature and vacuum level.

Therefore, its structure is very simple as an industrial concentration apparatus, and it is able to be used as

an economical apparatus having high treatment capacity with extremely easy operation.

Followings are description of examples of concentration operation using the concentration apparatus of the invention.

#### Example 1

A polymer solution which comprises 15 weight percent of copolymer comprising 92 % of acrylonitril, 7 % of vinyl acetate, and 1 % of methyl acrylate, and 85 weight percent of di-methyl-formaldehyde was stored in a tank at 80 °C, and concentration was done using an apparatus shown in Figure 1. The solution was sent to the concentration tank 5 with a gear pump 2 through triple layer tube heating unit 3. The solution passed through the heating unit 3 in 76 seconds in average and its temperature was 140 °C. Then the solution was sprayed into a concentration chamber 5 that was kept at 20 Torr of vacuum through a nozzle 4 of  $D = 1$  dia.,  $L/D = 10$ , and evaporated di-methyl formaldehyde was exhausted through 7 and condensed at 8. The solution taken out from the concentration chamber had average 300 seconds after passing through the heating unit, and its composition was polymer 18.6 fi and dimethyl formaldehyde 81.4 %. By above described method, the polymer solution was heat concentrated in as short as 6 minutes 7 seconds and no coloring or degradation was observed.

#### Example 2

Two types of polymer solutions were prepared which were 18 weight percent of copolymer comprising 92 % of acrylonitril, 7 % of vinyl acetate, and 1 % of methyl acrylate and 82 weight percent of di-methyl-formaldehyde, and 20 weight percent of said copolymer and 80 weight percent of di-methyl-formaldehyde, and they were fed by the gear pump 2 that was used in the Example 1. The feeding was done by alternatively switching said two polymer solutions which are different in composition for every batch. Fed polymer solution is heated up to 140 °C with a triple tube heater 3 while pressurizing so that evaporation of the solvent would not occur, then immediately splayed through a spray nozzle 4 into a concentration chamber 5 which is kept at 20 Torr. As a result, the concentration of the concentrated material, which had turned into having 23.5 weight percent of concentration of concentrated material, and in case of the one which had 20 weight percent of polymer concentration in fed polymer solution, polymer concentration of obtained concentrated material was 26.8 weight percent.

Further, a viscosity meter 13 for measuring the viscosity of concentrated-liquid was equipped at the section after the concentration chamber 5, and by this viscosity measurement, the temperature and pressure at the concentrating-liquid heating and pressurizing section was controlled so that the polymer concentration in the obtained concentrated-liquid is 25 weight percent, while keeping the pressure at the concentration chamber at 20 Torr. At first, the supply of concentration liquid with polymer concentration 18 weight percent was initiated and when the polymer concentration of concentrated-liquid reached 25 weight percent, supplied concentration liquid was switched to the one having 20 weight percent of polymer concentration, and the time required until the concentration of polymer in obtained concentrated-liquid becomes constantly 25.0 weight percent was measured, it took 10 minutes. And then the supplied

concentration liquid was switched to the one having polymer concentration 18 of weight percent, the polymer concentration in obtained concentrated-liquid went down to 24.6 weight percent at minimum and time needed until constantly obtaining 25.0 weight percent was about 10 minutes. In this case, the residence time of the concentrating-liquid at the pressurizing and heating section was 43 seconds, the residence time in the concentration chamber was 195 seconds. Obtained concentrated-liquid was not observed with coloring or generation of gels.

#### Example 3

600 parts of dimethyl-acetoamide was added to 220 parts of a wet polymer that comprised 100 parts of copolymer comprising acrylonitril 95 % and methyl acrylate 5 % and obtained by water precipitation polymerization and 120 parts of water, and made into slurry. When this slurry was separated for solid and liquid with a continuous centrifugal dehydrator, a wet polymer that is composed of 100 parts of polymer, 20 parts of water and 100 parts of dimethyl-acetoamide was continuously obtained. To the obtained wet polymer 100 parts, 310 parts of dimethylamide was added and heated to 50 °C to make it uniform solution and it was supplied to the concentration apparatus that was used in the Example 1. The polymer concentration and the water concentration of this concentrating-liquid were maintained within a range of  $19.6 \pm 1.1$  weight percent and  $3.9 \pm 0.3$  weight percent respectively. This concentrating-liquid was pressurized so that evaporation of solvent and water would not occur, and it was heated at the pressurizing and heating section up to 145 °C then sprayed into the concentration chamber that was kept at 19 Torr of pressure through the outlet 4 to have water and solvent evaporate to obtain concentrated-liquid, and concentrated-liquid of polymer concentration  $25.9 \pm 2.0$  weight percent and water  $0.8 \pm 0.12$  weight percent was constantly obtained.

Further, as same as the Example 2, when said concentrating-liquid was supplied by using a continuous concentration apparatus that is set to control the temperature of the pressurizing and heating section that is necessary to obtain concentrated-liquid at constant concentration by installing a line viscosity meter on the line after the concentration chamber to measure the polymer concentration in the concentrated-liquid by measuring the viscosity of the concentrated-liquid, it obtained the polymer concentration and water content of  $25.9 \pm 0.16$  weight percent and  $0.8 \pm 0.03$  weight percent respectively of the obtained concentrated-liquid, although the temperature of the concentrating-liquid that passes through the heating and pressurizing section varied within a range of  $145 \pm 19$  °C.

#### 4. Brief explanation of drawings

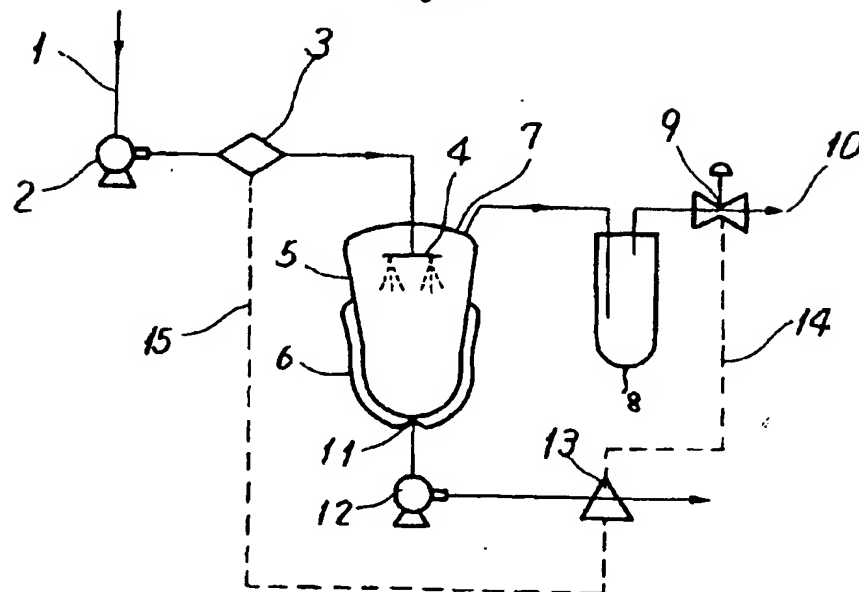
Figure 1 shows an example of schematic drawing of the concentration apparatus of this invention.

1. concentrating-liquid supply line
2. concentrating-liquid pressure feed pump
3. concentrating-liquid pressurizing and heating section
4. spray section

- 5 concentration chamber
- 6 concentration chamber temperature control unit
- 7 volatile component outlet
- 8 volatile component condensation section
- 9 concentration chamber pressure control unit
- 10. connection piping to pressure control source
- 11. concentrated-liquid outlet
- 12. concentrated-liquid takeout pump
- 13. concentrated-liquid analyzer
- 14. concentration chamber pressure control system
- 15. concentrating-liquid heat and pressure control system

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Figure 1



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④濃縮装置

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明 細 書

1. 発明の名称

濃 縮 装 置

2. 特許請求の範囲

- (1) 被濃縮液の加圧加熱部(A)と、加温加圧した被濃縮液噴霧部、濃縮室の圧力調整具、濃縮室の温度調整具、揮発成分排出口及び濃縮液取出口を備えた濃縮室(B)とをパイプラインで結合した濃縮装置。
- (2) 被濃縮液中の液体の蒸発潜熱を被濃縮液の溶液の顕熱に変え得る構造とした加圧加熱部(A)としたことを特徴とする特許請求の範囲第1項記載の濃縮装置。
- (3) 被濃縮液の加圧加熱部(A)と、加温加圧した被濃縮液噴霧部、濃縮室の圧力調整具、濃縮室の温度調整具、揮発成分排出口及び濃縮液取出口に備えた濃縮室(B)とをパイプラインで結合した濃縮装置であつて、濃縮液の組成分析具を組

込み、その分析結果より、加圧加熱部(A)の圧力温度並びに濃縮室の圧力を制御する制御系を組み込んだことを特徴とする濃縮装置。

- (4) 濃縮液の組成分析具がライン粘度計であることを特徴とする特許請求の範囲第3項記載の濃縮装置。
- (5) 被濃縮液中の液体の蒸発潜熱を被濃縮液の溶液の顕熱に変換するような構造とした加圧加熱部(A)としたことを特徴とする特許請求の範囲第3項記載の濃縮装置。

3. 発明の詳細な説明

本発明は低濃度の溶液を高濃度のものに濃縮するための装置に関するものであり、とくに多成分混合溶液であつてその粘度が中程度の溶液を濃縮するのに有効な濃縮装置に関するものである。

従来より溶液の濃縮装置は種々のものが開発されているが、それらの特徴は取扱う被濃縮液の粘度によつて装置の構造が異な



るようになっており、被濃縮液の粘度が高いものを濃縮する装置としては一軸蒸発装置、二軸蒸発装置、薄膜流下式蒸発器などが知られており、被濃縮液の粘度が低いものを濃縮する装置としては蒸発缶、多重効用缶などが知られている。しかしながら被濃縮液の粘度が $1 \sim 1,000$ ポイズ程度の中程度のものは、それ自体の挙動が極めて複雑であり取扱いにくいものが多く、効率的で汎用性のある濃縮装置は開発されておらず、個々の被濃縮液の物性、流動挙動を測定し、それぞれの被濃縮液に合った装置を個々に開発する必要があるのが現状である。

そこで本発明者等は粘度が中程度の被濃縮液を効率よく濃縮することが可能であり、また汎用性のある濃縮装置を開発することを目的として検討中のところ被濃縮液中の揮発すべき成分の蒸発潜熱を被濃縮液の溶液顕熱に変換した被濃縮液を低温低圧に保たれた濃縮室中に噴霧する方法を用いるこ

持する。3は被濃縮液加熱加圧部であり、被濃縮液中の除去すべき揮発成分の蒸発潜熱を顕熱として被濃縮液中に内蔵させる装置である。この加熱部の加熱装置としては、例えばスリットヒータ、オンレータ等々迅速に加熱しうる装置を用いることが望ましい。4は揮発成分の蒸発潜熱を内蔵させた被濃縮液の拡散面積を大きくさせ、自力で沸騰した揮発性成分を抵抗なく留去せしめるための噴霧部であり、被濃縮液を粉霧状態にするためにはノズル、スリット等々からなっていることが好ましい。5は濃縮室であり、6は濃縮室温度調整具であり、7は揮発性成分排出口であり、8は揮発性成分凝縮部である。9は濃縮室の真空度を制御するための圧力調整弁であり、10はその真空源につながる。11は濃縮溶液の取出口、12は濃縮溶液の吐出用ポンプであり、13は濃縮液の組成分析機器である。14は濃縮溶液の組成の分析結果より濃縮液の真空度を制御することにより濃縮溶液

とによつて比較的容易に濃縮液が得られることを見出し、このような方法に有効に利用しうる装置は被濃縮液の加熱加圧部と濃縮室とを分離した状態で保つ構造とすることが最も効率的な装置となることを見出し本発明を完成した。

本発明の要旨とするところは、被濃縮液の加熱加圧部(A)と被濃縮液の噴霧部、濃縮室の圧力調整装置、濃縮室の温度調整装置、揮発成分排出口及び濃縮液取出口を備えた濃縮室(B)とをパイプラインにて結合した濃縮装置並びに濃縮液の組成析器を設け、この析器を加圧加熱部(A)および又は、濃縮室の圧力調整装置をリレーによつて結合し被濃縮液の温度、圧力並びに濃縮室の圧力を調整するようにした濃縮装置にある。

第1図は典型的な本発明の濃縮装置の概略図である。第1図中1は被濃縮液供給路であり、2は被濃縮液加圧ポンプであり、このポンプにより被濃縮液を加圧状態に保

の組成を制御するための制御系である。15は濃縮溶液の組成分析結果より被濃縮部の加熱温度を制御し、濃縮溶液の組成を制御するための制御系である。濃縮室5は攪拌翼や掻き取り翼を備えることも可能であるが、本装置は加熱部と蒸発部が分離されているので濃縮槽壁への附着はほとんどないので特に必要としない。

本発明に於て用いる濃縮液分析器としては物理的又は化学的分析装置等種々のものを用いるが、最も簡便なのは揮発性成分を分析するのに便利な赤外線分析器であり又、物理的手段によるものとしては粘度計である。

また本発明の装置を用いて被濃縮液を濃縮するに際して注意すべきことは被濃縮液を加熱加圧する際に、被濃縮液中に含まれる物質が、加熱面でこげつきやゲル化などの濃度斑を生じさせないようにすることであり、このためには被濃縮液中の揮発性成分の蒸発潜熱を加熱加圧部に於て被濃縮液

の顕熱に変換せしめ、被濃縮液が濃縮室に噴霧されたときに揮散するようにせしめることである。

従来開発されてきた濃縮装置はこのような操作を施すことが難しいため、その濃縮過程において不都合な濃度分布が生ずるのであるが本発明の装置を用いる場合には、被濃縮液の加熱加圧部(A)と濃縮室とが分離されているので上述した如き不都合が生ずることはなく、また従来の装置を用いた場合に比べその濃縮時間を極めて短縮することが可能である。

本発明の濃縮装置によつて最も効率よく濃縮を行なうことができるものとしてはポリエステル<sup>1</sup>の初期重合時におけるエチレングリコールの除去による濃縮、水を含んだゴム組成物より水の除去による濃縮、水溶液重合によつて得た含水状態の重合体よりの水の除去による濃縮や湿式紡糸時に生ずるクズ糸より紡糸液を作る際の濃縮、とくに含水状態の重合体に対し、溶剤を加え、

(4) 蒸発時間が極小であるので精度よい制御を迅速に行ないうること。

(5) 蒸発面積を多大に取り得るので加熱温度・真空度だけで蒸発量を制御できると。

等々である。従つて工業用濃縮装置としては、構造が非常に簡単であり、操作的にも極めて容易な高処理能力を持つ経済的な装置として使用できる。

以下に本発明の濃縮装置を用いて濃縮を行なつた場合の例を以下に示す。

#### 実施例 1

アクリロニトリル 92%、酢酸ビニル 7%、アクリル酸メチル 1% からなる共重合体 15 重量%、ジメチルホルムアミド 85 重量% よりなる重合体溶液を 80℃ でタンクに貯留し第 1 図に示した装置によつて濃縮を行つた。該溶液をギアポンプ 2 で三重管加熱器 3 を通過せしめ濃縮槽 5 に送る。該溶液は平均的に 67 秒で加熱器 3 を通過しその温度は 140℃ であつた。引き続いて該溶液を 20 トルの真

水と溶剤の一部を揮散せしめて濃縮した重合体の溶剤溶液を作る場合などに利用することができる。

この場合、濃縮室(B)の圧力及び温度は、加熱加圧した被濃縮物を噴霧した場合揮発性成分が、例えば常沸状態で揮散するように選定することが必要である。

この濃縮装置の特徴を機能的に見れば、

- (1) 揮発成分を蒸発させるに必要な熱量を溶液の顕熱として時前に被濃縮溶液に内蔵させることができること。
- (2) 被濃縮溶液の顕熱として内蔵させた熱容量を揮発性成分の蒸発潜熱として使用し、被濃縮液を低温・低圧下に吐出させた時に揮発性成分が自己沸騰した際に効率よく拡散除去できるように拡散面積を充分に確保できるようにノズル、スリット等の細孔吐出部を備えていること。
- (3) 加熱機能と蒸発機能が完全に分離され、加熱部と蒸発部は別々に構成されているので濃縮物中に異物の生成がないこと。

空に保持された濃縮槽 5 に、 $D = 1/10$  のノズル 4 を通して噴出させ揮散したジメチルホルムアミドは 7 より排出し 8 にて凝縮した。濃縮槽から取出される溶液は加熱器を通過してから平均的に 300 秒経つておりその組成は重合体 1.6%、ジメチルホルムアミド 98.4% であつた。上記方法により、重合体溶液はわずか 6 分 7 秒で加熱濃縮されたことになり重合体の着色、変質は認められなかつた。

#### 実施例 2

アクリロニトリル 92%、酢酸ビニル 7%、アクリル酸メチル 1% からなる共重合体 15 重量%、ジメチルホルムアミド 85 重量% および前記共重合体 20 重量%、ジメチルホルムアミド 80 重量% の二種の重合体溶液を用基し、実施例 1 で用いた本装置のギアポンプ 2 により供給する。供給は 1 バッチ毎前述の組成の異なる二種の重合体溶液を順次切り変えて供給した。供給された重合体溶液を三重管加熱器 3 で溶剤の揮散が起らないように加圧

しながら、40℃に加熱し、直ちに20トルに保たれた濃縮室に噴霧口々を通じて噴出させる。その結果、供給重合体溶液中の重合体濃度が1.8重量%ものは濃縮物の濃度は2.35重量%となっており、供給重合体溶液中の重合体濃度が2.0重量%のものの場合には得られる濃縮物中の重合体濃度は2.68重量%であつた。

また濃縮室の後の部分に濃縮液の粘度を測定するための粘度計13を備え、この粘度測定により、得られる濃縮液中の重合体濃度が2.5重量%になるように濃縮室の圧力を20トルに保ち、被濃縮液の加温加圧部の温度圧力を調整した。まず初めに重合体濃度が1.8重量%の被濃縮液の供給を開始し、濃縮液の重合体濃度が2.5重量%のものとなるようになった時点で、供給する被濃縮液を重合体濃度が2.0重量%のものに切り換え、得られる濃縮液の重合体濃度が2.50重量%のものに定常的になるようになるまでの時間を測定したところ約10分間かかった。また次に

供給する被濃縮液を重合体濃度が1.8重量%のものに変えたところ、得られる濃縮液の重合体濃度は2.46重量%を最小値とし、2.50重量%のものが定常的に得られるようになるまでの所要時間は約10分であつた。この場合の被濃縮液の加圧加熱部滞在時間は43秒、濃縮室滞在時間は195秒であつた。得られた濃縮液は着色やゲルの生成は認められなかつた。

#### 実施例3

水析出重合によつて得たアクリロニトリル95%、アクリル酸メチル5%からなる共重合体100部、水120部よりなる湿潤重合体220部に、ジメチルアセトアミド600部を加えスラリー化する。このスラリーを連続遠心脱水機により固液分離すると重合体100部、水20部、ジメチルアセトアミド100部で構成される湿潤重合体が連続的に得られた。得られた湿潤重合体100部に対しジメチルアセトアミド310部を加えたものを50℃に加熱して均一溶液とし、実施例

1で用いた濃縮装置に供給した。この被濃縮液の重合体濃度は1.96±1.1重量%、水濃度は3.9±0.3重量%なる範囲に保たれていた。この被濃縮液の溶剤、水の揮散が起らないように加圧し、加圧加熱部で45℃に加熱したものを吐出口々を通じ、圧力19トルに保たれた濃縮室に噴霧し、水、溶媒を揮散せしめることによつて濃縮液を得たところ、重合体濃度2.59±0.2重量%、水0.8±0.12重量%の濃縮液が定常的に得られた。

また、実施例2と同様にして濃縮室の後の回路にライン粘度計を組み込み、濃縮液の粘度測定により濃縮液中の重合体濃度を測定し、一定濃度の濃縮液を得るのに必要な加圧加熱部の温度を制御するように調整した連続式濃縮器を用い上記被濃縮液を供給したところ、加熱加圧部を通過する被濃縮液の温度は45±1.9℃の範囲で変動したが、得られた濃縮液の重合体濃度は2.59±0.16重量%、水は0.8±0.03重量%の範囲のものが得られていた。

#### 図面の簡単な説明

第1図は本発明の濃縮装置の概略図の一例を示したものである。

- 1 被濃縮液供給路
- 2 被濃縮液供給加圧ポンプ
- 3 被濃縮液加圧加熱部
- 4 噴霧部
- 5 濃縮室
- 6 濃縮室温度調整具
- 7 揮発成分排出部
- 8 揮発成分凝縮部
- 9 濃縮室圧力調整具
- 10 圧力調整源への結合パイプ
- 11 濃縮液取出口
- 12 濃縮液吐出ポンプ
- 13 濃縮液分析器
- 14 濃縮室圧力制御系
- 15 被濃縮液加温、加圧度制御系

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図一

